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0227662.4

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Filtrona International Limited, 110 Park Street, LONDON.

NOV 2001

Patents ADP number (if you know it) 07617996 001

If the applicant is a corporate body, give the country/state of incorporation

GB

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Title of the invention

Activated Carbon Cigarette Filters

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Reddie & Grose 16 Theobalds Road LONDON WC1X 8PL

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DUPLICATE

TOBACCO SMOKE FILTER

This invention relates to tobacco smoke filters containing particulate sorbent.

Such use of sorbent particles to remove vapour phase (VP) components from tobacco smoke is well Cigarettes containing volatile flavourant (e.g. menthol) are also well known. However, prior attempts to use both volatile flavourant and particulate sorbent in a filter cigarette have been unsuccessful, it having impossible to provide a satisfactory level of flavour delivery whilst maintaining a satisfactory level of VP constituent removal by the particulate sorbent.

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The present invention provides a tobacco smoke filter containing activated carbon in which (1) pores of under 2 nm pore diameter (micropores) provide a pore volume of at most 0.3 cm³/g (N₂); and (2) (a) pores of 2 to 50 nm pore diameter (mesopores) provide a pore volume of at least 0.25 cm³/g (N₂) and/or (b) pores of 7 to 50 nm diameter (larger mesopores) provide a pore volume of at least 0.12 cm³/g (Hg). Herein a pore volume expressed in cm³/g (N₂) means said volume as measured by nitrogen porosimetry (using a Micromeritics Tristar 3000 for measurement of the nitrogen adsorption/desorption isotherms and characterising the pore

size distribution via the BJH method on the desorption branch of the isotherm. A pore volume or surface area expressed in cm<sup>3</sup>/g (Hg) or m<sup>2</sup>/g (Hg) means said value as measured by mercury porosimetry using a contact angle of 140° and a surface tension value of 480 dynes/cm.

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In the activated carbon used according to the invention pores of over 50 nm pore diameter (macropores) preferably provide a pore surface area of at least 5  $m^2/g$  (Hg), most preferably of 6 or more  $m^2/g$  (Hg).

The designation of pores of less than 2 nm, 2 to 50 nm, and over 50 nm size as micro-, meso- and macro-pores is in accord with accepted IUPAC terminology and definition.

The pore volume provided by said micropores is preferably at most  $0.26 \text{ cm}^3/\text{g}$  (N<sub>2</sub>), more preferably  $0.15 \text{ cm}^3/\text{g}$  (N<sub>2</sub>) or less. The pore volume provided by said 2 to 50 nm mesopores may for example be about  $0.3 \text{ cm}^3/\text{g}$  (N<sub>2</sub>) and is preferably over 0.4 or over  $0.5 \text{ cm}^3/\text{g}$  (N<sub>2</sub>); the preferred range is thus from 0.3 to 0.5 or higher cm<sup>3</sup>/g (N<sub>2</sub>). The pore volume provided by the 7 to 50 nm larger mesopores is preferably  $0.13 \text{ cm}^3/\text{g}$  (Hg) or higher, and can be over  $0.3 \text{ or over } 0.5 \text{ cm}^3/\text{g}$  (Hg); the preferred range is thus from 0.13 to  $0.5 \text{ or higher cm}^3/\text{g}$  (Hg).

We have most unexpectedly found that activated carbon of such carefully controlled micro/meso porosity - and preferably micro/meso/macro porosity - (a) shows satisfactory level of adsorption of volatile flavourant such as menthol (not too little and not too much); (b) releases sufficient of the flavour under smoking conditions to deliver satisfactory taste; (c) shows good adsorption of VP components from tobacco smoke; and (d) retains satisfactory (albeit reduced) level of this VP removal even in the presence of volatile flavourant such as menthol. This combination of properties has not heretofore been attainable.

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Accordingly the invention also provides a tobacco smoke filter according to the invention incorporated in a filter cigarette containing volatile flavourant - e.g. menthol. Such a filter cigarette provides for the first time the combination of flavour delivery to give an acceptably flavoured taste with an acceptable reduction in delivery of . VP smoke components.

The filter according to the invention may be of any design previously proposed for particulate sorbent-containing tobacco smoke filters. For example the carbon may be dispersed throughout a filter plug, carried on the tow or fibres or sheet material which is gathered to form the plug; it may instead be adhered to one or more threads

which extend through the matrix of the filter plug or be adhered to the inner face of a wrapper around the filter plug; or it may form a bed sandwiched between a pair of plugs (e.g. of cellulose acetate tow) in a common wrapper. The carbon may be treated with the flavourant prior to filter production so that it acts as a carrier for the flavour and minimises migration of the flavour during Instead, the carbon could be used in a suitable filter in the unflavoured state, with the flavour being added to another part of the filter and/or to the cigarette with which the filter is used and/or to the filter cigarette The flavourant might be carried on a wrapper packaging. around a filter plug or on one or more threads through a filter plug, and such plug may be the plug which also carries the activated carbon or a separate plug.

Filters according to the invention may additionally include one or more particulate sorbents other than the activated carbon required by the invention (e.g. silica gel, or a different carbon), mixed with the carbon required by the invention and/or separate from this.

The invention is illustrated by the following Examples, in which Examples B, C and D are according to the invention and the remainder are comparisons.

#### EXAMPLES

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For each example a sample of the respective activated carbon was dried and exposed to a menthol atmosphere in a desiccator at 55°C for 4 days, and the increase in weight was 'Triple cavity' cigarette filters were then recorded. assembled, each containing 100 mg of the mentholated carbon in a packed bed between two cellulose acetate filter The filter cigarettes were smoked under ISO conditions (35 cm3 puffs, each of two seconds duration, taken once per minute) and the menthol yields from the cigarettes were measured. The vapour phase of cigarette smoke was also collected and the percentage reduction of a selected number of vapour phase compounds measured; the mean reduction in these VP compounds, and the reduction obtained from an equivalent filter with 100 mg of the same carbon prior to exposure to menthol, were measured relative to an equivalent filter cigarette with no carbon.

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The results are summarised in the following Table which gives the porosity parameters for the various carbons employed and the measured performances of the filters using them. Examples B, C and D use activated carbons according to the requirements of the invention, whilst the remainder do not. Comparison Example A used a standard coconut-based carbon as typically used in prior cigarette filters, whilst Comparison Examples E to G used other carbons whose micro/meso/macro porosity led to poor results.

## TABLE

EXAMPLE	Æ	В	ບ	Q	函	Ē	ŋ
Micropore Volume (N <sub>2</sub> ) - (cm <sup>3</sup> /g)	0.46	0.26	0.11	0.12	0.52	0.57	0.23
$2-50$ nm Mesopore Volume $(N_2)$ -cm $^3/g$	0.09	0.30	0.44	0.51	0.36	0.25	0.04
7-50nm Mesopore Volume (Hg)-cm³/g	90.0	0.13	0.34	0.54	0.21	0.15	na *
Macropore Area (Hg)-m²/g	1.9	6.4	6.9	12.2	1.4	4.9	na *
Menthol Uptake %	18.6	27.3	27.5	23	57.1	18.9	11.5
Menthol Yield (mg/cig)	0.03	0.73	0.44	0.72	0.07	0.06	0.15
Mean VP (unmentholated) (%)	53	85	45	61	85	45	47
Mean VP (mentholated) (%)	<5	24	24	36	<5	<5	<5

\* na - not ascertained

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### CLAIMS:

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- 1. A tobacco smoke filter containing activated carbon in which micropores of under 2 nm pore diameter provide a micropore volume of at most 0.3 cm $^3$ /g (N $_2$ ) and mesopores of 2 to 50 nm pore diameter provide a mesopore volume of at least 0.25 cm $^3$ /g (N $_2$ ).
- 2. A tobacco smoke filter containing activated carbon in which micropores of under 2 nm pore diameter provide a micropore volume of at most 0.3 cm $^3$ /g (N $_2$ ) and mesopores of 7 to 50 nm pore diameter provide a mesopore volume of at least 0.12 cm $^3$ /g (Hg).
- 3. A tobacco smoke filter containing activated carbon in which micropores of under 2 nm pore diameter provide a micropore volume of at most 0.3 cm $^3$ /g (N $_2$ ), mesopores of 2 to 50 nm pore diameter provide a mesopore volume of at least 0.25 cm $^3$ /g (N $_2$ ), and mesopores of 7 to 50 nm pore diameter provide a mesopore volume of at least 0.12 cm $^3$ /g (Hg).
- 4. A filter according to any preceding claim wherein macropores of over 50 nm diameter provide a surface area of at least 5  $m^2/g$  (Hg).

- 5. A filter according to any preceding claim wherein said micropore volume is at most  $0.26 \text{ cm}^3/\text{g}$  (N<sub>2</sub>).
- 6. A filter according to any preceding claim wherein said micropore volume is at most 0.15 cm $^3/g$  ( $N_2$ ).
- 7. A filter according to any preceding claim wherein said . 2 to 50 nm mesopore volume is about 0.3 cm $^3/g$  (N<sub>2</sub>).
  - 8. A filter according to any of claims 1 to 6 wherein said 2 to 50 nm mesopore volume is over 0.4 or over 0.5 cm $^3/g$  (N<sub>2</sub>).
- 9. A filter according to any preceding claim wherein said 7 to 50 nm mesopore volume is at least 0.13 cm<sup>3</sup>/g (Hg).
  - 10. A filter according to any preceding claim wherein said 7 to 50 nm mesopore volume is over 0.3 or over 0.5 cm $^3/g$  (Hg).
- 15 11. A tobacco smoke filter substantially as described in Example B or C or D.
  - 12. A filter cigarette containing volatile flavourant and including a filter according to any preceding claim.

- 13. A filter cigarette according to claim 12 wherein said flavourant comprises menthol.
- 14. A filter cigarette according to claim 12 or 13 wherein said flavourant is applied to said activated carbon.
- 5 15. A filter cigarette according to any of claims 12 to 14 wherein said flavourant is applied to a part of said filter or cigarette other than said activated carbon and/or to packaging for said cigarette.

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